



ICF International / Laboratory Data Consultants

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MEMORANDUM

TO: Chris Lichens, Remedial Project Manager
Site Cleanup Section 4, SFD-7-4

THROUGH: Rose Fong, ESAT Task Order Manager (TOM) *RF*
Quality Assurance (QA) Program, MTS-3

FROM: Doug Lindelof, Data Review Task Manager *[Signature]*
Region 9 Environmental Services Assistance Team (ESAT)

ESAT Contract No.: EP-W-06-041
Technical Direction Form No.: 00105041 Amendment 6

DATE: July 9, 2007

SUBJECT: Review of Analytical Data, **Tier 2**

Attached are comments resulting from ESAT Region 9 review of the following analytical data:

Site:	Omega Chem OU2
Site Account No.:	09 BC LA02
CERCLIS ID No.:	CAD042245001
Case No.:	Not Provided
SDG No.:	IPH2899, IPH3042, IPH3163, IPI0246, IPI0558, IPI0711, and IPI0851
Laboratory:	Test America Analytical Testing Corp.
Analysis:	1,2,3-Trichloropropane (1,2,3-TCP) and n- Nitrosodimethylamine (NDMA)
Samples:	37 Water Samples (see Case Summary)
Collection Date:	August 28, 29, and 30, 2007 and September 5, 7, 8, and 11, 2006
Reviewer:	Santiago Lee, ESAT/Laboratory Data Consultants (LDC)

This report has been reviewed by the EPA TOM for the ESAT contract, whose signature appears above.

If there are any questions, please contact Rose Fong (QA Program/EPA) at (415) 972-3812.

Attachment

SAMPLING ISSUES: ☐ Yes ☒ No

Data Validation Report – Tier 2

Case No.: Not Provided
SDG No.: IPH2899, IPH3042, IPH3163, IPI0246, IPI0558, IPI0711, and IPI0851
Site: Omega Chem OU2
Laboratory: Test America Analytical Testing Corp.
Reviewer: Santiago Lee, ESAT/LDC
Date: July 9, 2007

I. CASE SUMMARY

Sample Information

SDG IPH2899 Samples: OC2-MW18C-W-2-208, OC2-MW18C-W-0-209, OC2-MW18B-W-0-210, OC2-MW18A-W-0-211, and OC2-MW7-W-0-212

SDG IPH3042 Samples: OC2-MW4C-W-0-214, OC2-MW4B-W-0-215, OC2-MW4B-W-1-216, OC2-MW4A-W-5-217, OC2-MW5-W-0-218, and OC2-MW15-W-0-219

SDG IPH3163 Samples: OC2-MW8D-W-2-221, OC2-MW8D-W-0-222, OC2-MW8C-W-0-223, OC2-MW8B-W-0-224, OC2-MW8A-W-0-225, and OC2-MW6-W-0-226

SDG IPI0246 Samples: OC2-MW17C-W-2-241, OC2-MW17C-W-0-243, OC2-MW17B-W-5-244, and OC2-MW17A-W-0-245

SDG IPI0558 Samples: OC2-MW19-W-0-246, OC2-MW11-W-0-247, OC2-MW3-W-0-248, OC2-MW10-W-0-249, OC2-MW10-W-1-250, and OC2-MW2-W-0-251

SDG IPI0711 Samples: OC2-MW13B-W-0-253, OC2-MW12-W-0-254, OC2-MW1B-W-0-255, OC2-MW1A-W-0-256, and OC2-MW1A-W-2-257

SDG IPI0851 Samples: OC2-MW23D-W-0-259, OC2-MW23D-W-0-260, OC2-MW23C-W-0-261, OC2-MW23C-W-1-262, and OC2-MW14-W-0-263

Concentration and Matrix: Low Concentration Water

Analysis: 1,2,3-TCP (GC/MS) and NDMA (GC/MS/MS CI)

Method: EPA Methods 524.2 and 1625 Modified

Collection Date: August 28, 29, and 30, 2007 and September 5, 7, 8, and 11, 2006

Sample Receipt Date: August 28, 29, and 30, 2007 and September 5, 7, 8, and 11, 2006

Extraction Date: August 30 and 31, 2006 and September 6, 7, 8, 11, 12, 13, and 14, 2006

Analysis Date: August 30, 2006 and September 5, 6, 7, 11, 12, 13, 15, 16, and 18, 2006

Field QC

Field Blanks (FB): OC2-MW18C-W-2-208, OC2-MW8D-W-2-221, OC2-MW17C-W-2-241

Trip Blanks (TB): Not Provided

Equipment Blanks (EB): Not Provided

Background Samples (BG): Not Provided

Field Duplicates (D1): OC2-MW4B-W-0-215 and OC2-MW4B-W-1-216

Field Duplicates (D2): OC2-MW10-W-0-249 and OC2-MW10-W-1-250

Field Duplicates (D3): OC2-MW23C-W-0-261 and OC2-MW23C-W-1-262

Laboratory QC

Method Blanks & Associated Samples:

6H31162-BLK1: (NDMA) OC2-MW18C-W-0-209, OC2-MW18B-W-0-210, OC2-MW18A-W-0-211, OC2-MW7-W-0-212
6I11078-BLK1: (NDMA) OC2-MW4C-W-0-214, OC2-MW4B-W-0-215, OC2-MW4B-W-1-216, OC2-MW4A-W-5-217, OC2-MW5-W-0-218, OC2-MW15-W-0-219
6I06059-BLK1: (NDMA) OC2-MW8D-W-0-222, OC2-MW8C-W-0-223, OC2-MW8B-W-0-224, OC2-MW8A-W-0-225, OC2-MW6-W-0-226
6I12054-BLK1: (NDMA) OC2-MW17C-W-0-243, OC2-MW17B-W-5-244, OC2-MW17A-W-0-245; OC2-MW19-W-0-246; OC2-MW23C-W-1-262
6I14061-BLK1: (NDMA) OC2-MW11-W-0-247, OC2-MW3-W-0-248, OC2-MW10-W-0-249, OC2-MW10-W-1-250, OC2-MW2-W-0-251; OC2-MW13B-W-0-253, OC2-MW12-W-0-254, OC2-MW1B-W-0-255, OC2-MW1A-W-0-256; OC2-MW23D-W-0-259, OC2-MW23D-W-0-260, OC2-MW23C-W-0-261, OC2-MW14-W-0-263
C6H3002-BLK1: (1,2,3-TCP) OC2-MW18C-W-2-208, OC2-MW18C-W-0-209, OC2-MW18B-W-0-210, OC2-MW18A-W-0-211, OC2-MW7-W-0-212; OC2-MW4C-W-0-214, OC2-MW4B-W-0-215
C6I0601-BLK1: (1,2,3-TCP) OC2-MW4B-W-1-216, OC2-MW4A-W-5-217, OC2-MW5-W-0-218, OC2-MW15-W-0-219; OC2-MW8D-W-2-221, OC2-MW8D-W-0-222, OC2-MW8C-W-0-223, OC2-MW8B-W-0-224, OC2-MW8A-W-0-225
C6I0702-BLK1: (1,2,3-TCP) OC2-MW6-W-0-226
C6I1103-BLK1: (1,2,3-TCP) OC2-MW17C-W-2-241, OC2-MW17C-W-0-243, OC2-MW17B-W-5-244, OC2-MW17A-W-0-245
C6I1201-BLK1: (1,2,3-TCP) OC2-MW19-W-0-246, OC2-MW11-W-0-247, OC2-MW3-W-0-248, OC2-MW10-W-0-249, OC2-MW10-W-1-250, OC2-MW2-W-0-251; OC2-MW13B-W-0-253
C6I1302-BLK1: (1,2,3-TCP) OC2-MW12-W-0-254, OC2-MW1B-W-0-255, OC2-MW1A-W-0-256, OC2-MW1A-W-2-257; OC2-MW23D-W-0-259, OC2-MW23D-W-0-260, OC2-MW23C-W-0-261, OC2-MW23C-W-1-262, OC2-MW14-W-0-263

Tables

1B: Data Qualifier Definitions for Organic Data Review

Sampling Issues

The COC forms did not specify the samples to be used for laboratory quality control (QC). The laboratory selected samples OC2-MW4A-W-5-217, OC2-MW6-W-0-226, and OC2-MW1B-W-0-255 for 1,2,3-TCP matrix spike/matrix spike/duplicate (MS/MSD) analysis and selected sample OC2-MW17B-W-5-224 for NDMA MS analysis. The effect on data quality is not known.

Additional Comments

As directed by the EPA TOM, a Tier 2 data review was performed (review all QC results and calibrations, minus calculation check). A Table 1A is not requested.

For the NDMA analysis, decafluorotriphenylphosphine (DFTPP) was not analyzed. Since NDMA is analyzed by the chemical ionization (CI) technique, no adverse effect is expected.

This report was prepared in accordance with the following documents:

- ESAT Region 9 Standard Operating Procedure 901, *Guidelines for Data Review of Contract Laboratory Program Analytical Services (CLPAS) Volatile and Semivolatile Data Packages*;
- EPA Method 524.2, *Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry*, Revision 4.1, 1995;
- EPA Method 1625C, *Semivolatile Organic Compounds by Isotope dilution GC/MS*, June 1989; and
- USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, October 1999.

II. VALIDATION SUMMARY

The data were evaluated based on the following parameters:

<u>Parameter</u>	<u>Acceptable</u>	<u>Comment</u>
1. Holding Time/Preservation	No	B
2. GC/MS and GC Performance	Yes	
3. Initial Calibration	Yes	
4. Continuing Calibration	Yes	
5. Laboratory Blanks	Yes	
6. Field Blanks	Yes	
7. Surrogate (Method 524.2)	N/A	
8. Labeled Compound (Method 1625)	No	F
9. Matrix Spike/Matrix Spike Duplicates	No	D
10. Laboratory Control Samples/Duplicates	No	C
11. Internal Standard	Yes	
12. Compound Identification	N/A	
13. Compound Quantitation	No	A, E
14. System Performance	Yes	
15. Field Duplicate Sample Analysis	Yes	

N/A = Not Applicable

III. VALIDITY AND COMMENTS

A. The following detected results are qualified as estimated and should be flagged "J".

- NDMA in samples OC2-MW4A-W-5-217, OC2-MW5-W-0-218, OC2-MW15-W-0-219, OC2-MW17A-W-0-245, OC2-MW2-W-0-251, OC2-MW12-W-0-254, OC2-MW23C-W-0-261, OC2-MW23C-W-1-262, and OC2-MW14-W-0-263 (below the practical quantitation limit)

Results below the practical quantitation limits (PQLs) are considered to be qualitatively acceptable, but quantitatively unreliable, due to the uncertainty in analytical precision near the limit of detection.

B. Results for the following analytes are qualified as estimated due to missed technical holding times and should be flagged "J".

- NDMA in samples OC2-MW4C-W-0-214, OC2-MW4B-W-0-215, OC2-MW4B-W-1-216, OC2-MW4A-W-5-217, OC2-MW5-W-0-218, and OC2-MW15-W-0-219

The extraction of samples listed above exceeded the 7-day technical holding time for water samples as shown below.

<u>Sample</u>	<u>Date Collected</u>	<u>Date Extracted</u>	<u># of Days Exceeded</u>
OC2-MW4C-W-0-214	08/29/06	09/11/06	6
OC2-MW4B-W-0-215	08/29/06	09/11/06	6
OC2-MW4B-W-1-216	08/29/06	09/11/06	6
OC2-MW4A-W-5-217	08/29/06	09/11/06	6
OC2-MW5-W-0-218	08/29/06	09/11/06	6
OC2-MW15-W-0-219	08/29/06	09/11/06	6

Detected results for the samples listed above may be biased low. Where results are nondetected, false negatives may exist.

- C. For the NDMA analysis, the laboratory control sample/laboratory control sample duplicate (LCS/LCSD) relative percent difference (RPD = 21%) for QC batch 6I14061 (SDGs IPI0246, IPI0558, IPI0711, and IPI0851) did not meet the laboratory QC limit of 0-20%. Results reported may indicate poor laboratory technique. The effect on data quality is not known.
- D. For the 1,2,3-TCP analysis, the MS/MSD RPD (29%) for sample OC2-MW1B-W-0-255 (SDG IPI0711) did not meet the laboratory QC limit of 0-20%. The result reported may indicate poor laboratory technique or matrix effects which may interfere with analysis. The effect on data quality is not known.
- E. The laboratory reported the NDMA sample practical quantitation limit (PQL) as 0.0019 ug/L. However, areas for the low standard are only 2049 and 843 for 08/02/06 and 09/12/06 initial calibrations, respectively (see attached quantitation

reports, p. 25 in IPH2899 and p. 28 in IPH3042 data packages). Furthermore, areas for NDMA detected results are relatively low. For example, the area is only 1669 for the concentration of 0.0019 ug/L in sample OC2-MW4A-W-5-217 (see attached quantitation report, p. 131 in IPH3042 data package). In the reviewer's professional judgment, the sample PQL should be raised to 0.01 ug/L; non-detected sample results should be reported as 0.01U.

- F. For the NDMA analysis, the laboratory did not spike the samples and method blanks with a labeled compound (i.e., surrogate; see Method 1625C Sections 6.8, 10.2.1.3, and 10.2.3.2 and Figure 4). Consequently, the extraction efficiency (surrogate recovery) cannot be evaluated. The NDMA-d6 spiked by the laboratory was used as an internal standard.

TABLE 1B

DATA QUALIFIER DEFINITIONS FOR ORGANIC DATA REVIEW

The definitions of the following qualifiers are prepared according to the document, "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," October 1999.

- U The analyte was analyzed for but was not detected above the reported sample quantitation limit.
- L Indicates results which fall below the Contract Required Quantitation Limit. Results are estimated and are considered qualitatively acceptable but quantitatively unreliable due to uncertainties in the analytical precision near the limit of detection.
- J The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
- NJ The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
- UJ The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
- R The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

Quantitation Report (Not Reviewed)

Data File : C:\MSDCHEM\1\DATA\06SEP12\C0912013.D
 Acq On : 12 Sep 2006 10:55 pm
 Sample : IPH3042-04RE1
 Misc : WATER 1L/1mL ----- Batch 6I11078
 MS Integration Params: rteint.p
 Quant Time: Sep 13 6:45 2006

Vial: 24
 Operator: DF/AI
 Inst : gcms37
 Multiplr: 1.00

Quant Results File: C6I12NWA.RES

Quant Method : C:\MSDCHEM\1\METHODS\C6I12NWA.M (RTE Integrator)
 Title : Nitrosamine Water ICAL 9/12/06, Preextraction IS
 Last Update : Tue Sep 12 17:55:56 2006
 Response via : Initial Calibration
 DataAcq Meth : C6I12NWA

Internal Standards	R.T.	QIon	Response	Conc	Units	Dev(Min)
1) NDMA-D6	10.79	81	4169	10.00	PPB	0.00
4) NDPA-D14	15.22	145	2493	10.00	PPB	0.00
Target Compounds						
2) NDMA	10.73	92	1669	2.03	PPB	80
3) NDEA	12.97	120	157	0.36	PPB	86
5) NDPA	15.23	148	87	0.28	PPB	1
6) NPYR	16.45	118	29	0.13	PPB	67

Qvalue

(#) = qualifier out of range (m) = manual integration
 C0912013.D C6I12NWA.M Wed Sep 13 06:45:00 2006

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Quantitation Report (Not Reviewed)

Data File : C:\MSDCHEM\1\DATA\06AUG02\NNA001.D

Vial: 3

Acq On : 2 Aug 2006 12:47 pm

Operator: DF/AI

Sample : 1PPB WATER ICAL STD# 6060243

Inst : gcms37

Misc : n-Nitrosamines Water ICAL

Multiplr: 1.00

MS Integration Params: rteint.p

Quant Time: Aug 2 15:43 2006

Quant Results File: C6H02NWA.RES

Quant Method : C:\MSDCHEM\1\METHODS\C6H02NWA.M (RTE Integrator)

Title : Nitrosamine Water ICAL 8/02/06, Preextraction IS

Last Update : Wed Aug 02 15:42:46 2006

Response via : Initial Calibration

DataAcq Meth : C6F30NWA

Internal Standards	R.T.	QIon	Response	Conc	Units	Dev(Min)
1) NDMA-D6	10.78	81	4794	10.00	PPB	0.00
4) NDPA-D14	15.23	145	6315	10.00	PPB	0.00
Target Compounds						Qvalue
2) NDMA	10.73	92	2049	0.99	PPB	99
3) NDEA	12.98	120	1602	0.80	PPB	98
5) NDPA	15.19	148	1288	0.94	PPB	95
6) NPYR	16.47	118	1163	0.90	PPB	96

(#) = qualifier out of range (m) = manual integration

NNA001.D C6H02NWA.M Wed Aug 02 15:43:10 2006

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Quantitation Report (Not Reviewed)

Data File : C:\MSDCHEM\1\DATA\06SEP12\NNA001.D
 Acq On : 12 Sep 2006 2:44 pm
 Sample : 1PPB WATER ICAL STD# 6060243
 Misc : n-Nitrosamines Water ICAL
 MS Integration Params: rteint.p
 Quant Time: Sep 12 17:53 2006

Vial: 3
 Operator: DF/AI
 Inst : gcms37
 Multiplr: 1.00

Quant Results File: C6I12NWA.RES

Quant Method : C:\MSDCHEM\1\METHODS\C6I12NWA.M (RTE Integrator)
 Title : Nitrosamine Water ICAL 9/12/06, Preextraction IS
 Last Update : Tue Sep 12 17:53:07 2006
 Response via : Initial Calibration
 DataAcq Meth : C6H02NWA

Internal Standards	R.T.	QIon	Response	Conc	Units	Dev(Min)
1) NDMA-D6	10.78	81	3840	10.00	PPB	0.00
4) NDPA-D14	15.22	145	1282	10.00	PPB	0.00
Target Compounds						Qvalue
2) NDMA	10.73	92	843	0.61	PPB	98
3) NDEA	12.97	120	386	0.30	PPB	89
5) NDPA	15.18	148	180	0.81	PPB	71
6) NPYR	16.45	118	124	0.54	PPB	97

(#) = qualifier out of range (m) = manual integration
 NNA001.D C6I12NWA.M Tue Sep 12 17:53:21 2006

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